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Factors Influencing the Volatilization of Mercury from Soil

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FACTORS INFLUENCING THE VOLATILIZATION OF MERCURY FROM SOIL

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FOREWORD

Protection of the environment requires effective regulatory actions which are based on sound technical and scientific information. This information must include the quantitative description and linking of pollutant sources, transport mechanisms, interactions, and resulting effects on man and his environment. Because of the complexities involved, assessment of specific pollutants in the environment requires a total systems approach which transcends the media of air, water, and land. The Environmental Monitoring and Support Laboratory-Las Vegas contributes to the formation and enhancement of a sound monitoring data base for exposure assessment through programs designed to:

- develop and optimize systems and strategies for monitoring pollutants and their impact on the environment
- demonstrate new monitoring systems and technologies by applying them to fulfill special monitoring needs of the Agency's operating programs

A study was conducted to determine some of the factors responsible for the volatilization of mercury from soils amended with mercury. It was discovered that the amount of mercury lost from the soil could be correlated with the amount of mercury in the soil that is solubilized by ammonium nitrate. In addition it was found that the volatilization was mediated by microorganisms. The conclusions can be beneficial in designing experiments dealing with mercury compounds and soils and also in the interpretation of data gathered by other investigators. Users who should find the report of value include the Office of Air Programs, Office of Toxic Substances, laboratories within the Office of Research and Development, other Federal agencies, University and industrial research staffs.

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ABSTRACT

Mercury volatilization from soils amended to 1 ppm mercury with mercuric nitrate ceased within 1 week after application. During the first week, 20% of the applied mercury was lost from a silty clay-loam soil and 43% was lost from a loamy sand soil. Volatilization of Hg from the loamy sand soil resulted in a concurrent decrease in ammonium nitrate-extractable mercury. Other work with sterile soil indicates that the volatilization was mediated by microorganisms.

CONCLUSIONS

Mercury (Hg) when applied to soil in a soluble form was initially very rapidly volatilized from the soil. The rate of volatilization decreased with time until it approached zero even though the soil still contained 50 to 80% of the applied Hg. Increasing the concentration of soil Hg increased the period of maximum volatility.

Volatilization is the result of microbial action. The rate is determined by the availability of Hg to the microbes. This in turn is determined by many factors including soil texture and organic matter content.

RECOMMENDATIONS FOR FUTURE RESEARCH

Clarification of how Hg is bound in soil is needed. Also, information on the volatility of various species of Hg is needed to understand the complexities of Hg contamination of soil. It is likewise important to determine the microorganisms involved in these reactions in order to understand and predict mercury movement and its potential threat to food contamination.

INTRODUCTION

Interest in the environmental cycling of Hg has turned to the transformation of Hg into forms other than organics. It is known that Hg applied to the soil in many chemical forms can be lost as volatiles (Alberts et al., 1974; Kimura and Miller, 1964; Hitchcock and Zimmerman, 1957). However, no definitive study on the volatility rate, chemical species formed, and binding of mercury in soil has been reported. What mediates the volatilization of Hg from soil systems is not well understood. Kimura and Miller (1964) noted that autoclaved soils showed a decreased loss of Hg vapor, thus indicating a biological interaction. Specific microorganisms have been isolated from lake and river sediments (Izaki, 1977; Holm and Cox, 1975; Avotins and Jenne, 1975; Summers and Silver, 1972; Tonomura, Maeda, and Futai, 1968) which reduce ionic Hg to elemental Hg. These same microbes can be found in aerobic soil; therefore, it is reasonable to assume that there can be biological reduction and volatilization of Hg from natural and agricultural soils. The duration and rates of Hg volatilization after application are not available. In addition, the solubility of Hg in the soil solution as related to the volatility has not been determined.

A study was conducted to investigate the volatilization of Hg from amended soils, the rate of loss, the chemical and biological availability of Hg in soil, and whether such volatility is of a biological origin.

MATERIALS AND METHODS

Two soils, a loamy sand and a silty clay-loam, were used for this study. They were collected from the upper 10 centimeters (cm) of the Ap horizon (plow layer) in an agricultural area of southern Nevada. The moist soil was processed through a 2-millimeter (mm) sieve and stored at room temperature in plastic bags. Some physical and chemical properties of the soils are listed in Table 1. Sterilized soil was prepared by steam autoclaving 200-gram (g) quantities at 121° C for 4 hours. After 3 days the samples were reautoclaved. To insure that the soils were indeed sterile, 0.1 g of each soil was spread over the surface of nutrient agar in a petri dish. These samples were then incubated for 1 week. No growth was seen on the inoculated dishes.

TABLE 1. PHYSICAL AND CHEMICAL PROPERTIES OF SOIL

Soil (Texture Classification)	Series	Sand	Clay %	Organic Carbon	Cation Exchange Capacity meq/100g	pH
Sand (Loamy Sand)	Bluepoint - a member of the mixed thermic family of Typic Torripsamment	79.8	3.5	0.53	4.3	9.0
Clay (Silty Clay Loam)	Overton - a member of the fine montmorillonitic calcareous thermic family of Mollic Haplaquepts	14.7	34.4	3.44	29.0	7.8

Mercuric nitrate [$Hg(NO_3)_2$] was used to amend the soil. To each portion of soil (20 g), 20 micrograms (µg) of Hg was added and stirred. This resulted in a concentration of 1 part per million (ppm) Hg. Radioactive mercury [$^{203}Hg(NO_3)_2$] was mixed with the stable $Hg(NO_3)_2$ before the soil addition. Those soils from which volatile Hg was to be determined received 40 nanocuries (nCi) of ^{203}Hg and the soils used for extraction purposes received 4 nCi of ^{203}Hg. The mixed solution was such that 2 milliliters (ml) of solution contained the desired amount of both stable and radioactive Hg. The Hg solution was added to 20 g of soil contained in 250-ml polyethylene bottles. After addition, distilled water was added to bring the soil moisture to 50% of the soil moisture-holding capacity. The bottles with their contents were weighed daily and the amount of water lost through

evaporation was replaced as necessary. The bottles were closed with rubber stoppers containing connections for inlet and outlet air lines. All bottles were flushed during the study at a rate of 20 cubic centimeters per minute (cc/min) with water-saturated, compressed air.

Air flushed from the bottles being used for the volatility experiments was passed through a charcoal collector located inside a side-hole scintillation crystal. Flexible tubing used to connect the bottle to the collector had a polyethylene liner. The charcoal collector was made from a 10-ml volumetric pipette cut to length, with the bulb being filled with charcoal pulverized to a 16 mesh.

Air from bottles whose contents were to be used in the extraction portion of this study was passed through charcoal traps connected to the outlet tubes of the rubber stopper. These traps were used as a safety measure and not to determine volatile Hg. It was not necessary to change them during the course of the study.

Each of the two side-hole scintillation crystals was connected to a single-channel analyzer and the output was recorded on a strip chart recorder. In this way a continuous record was obtained of the amount of volatilization in terms of ^{203}Hg disintegrations as cpm. The charcoal collectors were changed 2 to 4 times a day during the first 72 hours of each experiment to insure that the cpm rate did not go off scale. The bottles were maintained at 25° C in water baths. Figure 1 is a representation of the experimental set up.

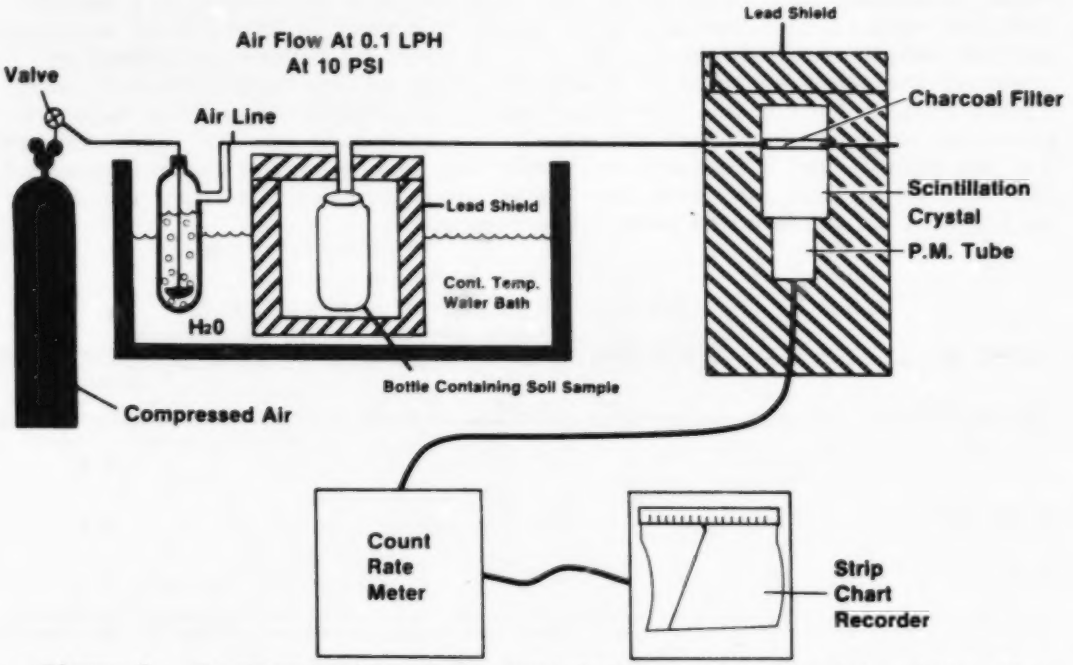

Figure 1. Representation of the experimental setup for determining the rate of Hg volatilization.

After an experiment, the counts per minute (cpm) which had been continously recorded on graph paper were converted to a more meaningful form by applying appropriate corrections for decay, specific activity, efficiency and unit conversion. The slope of the line was then determined at 1-hour intervals and the results presented as the Hg volatility rate (nanograms per hours, ng/h).

Soils amended with Hg were subjected to different extracting agents after 24 hours of incubation in order to determine one suitable for use. These solutions included H_2O, 1N NH_4NO_3, and DTPA (Follett and Lindsay, 1971). Each extraction was for 18 hours and included vigorous agitation on a rotary shaker. The soil mixture was then centrifuged and the supernatant was passed through a Whatman number 1 filter paper. The resulting clear extract was used for the ^{203}Hg determination. Table 2 gives the amount of the applied Hg which was extracted from each of the soils. In all subsequent experiments 1N NH_4NO_3 was used for the extractions.

Experiments were designed so that volatile mercury was determined from one set of replicates and the extractable and soil-bound mercury from another set. By combining these results a budget could be reconstructed. Agreement was generally good between the amount of mercury lost via volatilization as determined by integrating the loss-rate curve and the amount determined by subtracting the amount remaining as soil-bound and extractable from the original amount of the amendment. However, there was one exception where there was a discrepancy of 18% between these two methods of determining loss. Because of greater precision the results of the direct analysis of the volatile mercury are presented as the most reliable measure of the amount lost via this route. The amount of mercury in the 1N NH_4NO_3-wash solution and the amount remaining in the soil after extraction were determined by counting the gamma activity on a sodium iodide scintillation crystal using a multi-channel analyzer. For convenience in interpretation, the data are presented so that the volatility rate is superimposed over the data depicting the soluble and nonsoluble Hg. This was done to allow visualization of how changes in loss rate were correlated with amounts of mercury residual in the soil as soluble and nonsoluble compounds.

TABLE 2. PERCENT MERCURY EXTRACTED FROM SOIL WITH VARIOUS EXTRACTANTS AFTER 24-HOUR INCUBATION

Extractant	% Recovered from Sand	% Recovered from Clay
H_2O	8.5	0.6
1N NH_4NO_3	20.5	0.6
DTPA	8.2	0.6

RESULTS

Various combinations of soil type, Hg concentration, and sterile and natural soils were investigated. Replicates of experimental trials yielded similar results but varied slightly in summation of the mercury budgets. Yet all replicates gave similar rates, thus representative curves will be presented instead of the entire data collected. When sandy soils were amended with 20 µg Hg/20 g soil (1 ppm Hg), 43% of the applied mercury was lost within the first 6 days. The data presented in Figure 2 show that volatilization increased reaching a peak rate at about 40 hours after the Hg was added. The rate of decreasing 1N NH_4NO_3-soluble and nonsoluble mercury content was greatest when the volatility rate was at its maximum. Soluble Hg decreased from 30% of the amount initially applied to about 2% at the termination of these tests. The volatility rate decreased to a minimum by about 100 hours after which there appeared to be a steady state established. Some volatility continued at a rate which appeared to be in equilibrium with the amount of soluble Hg.

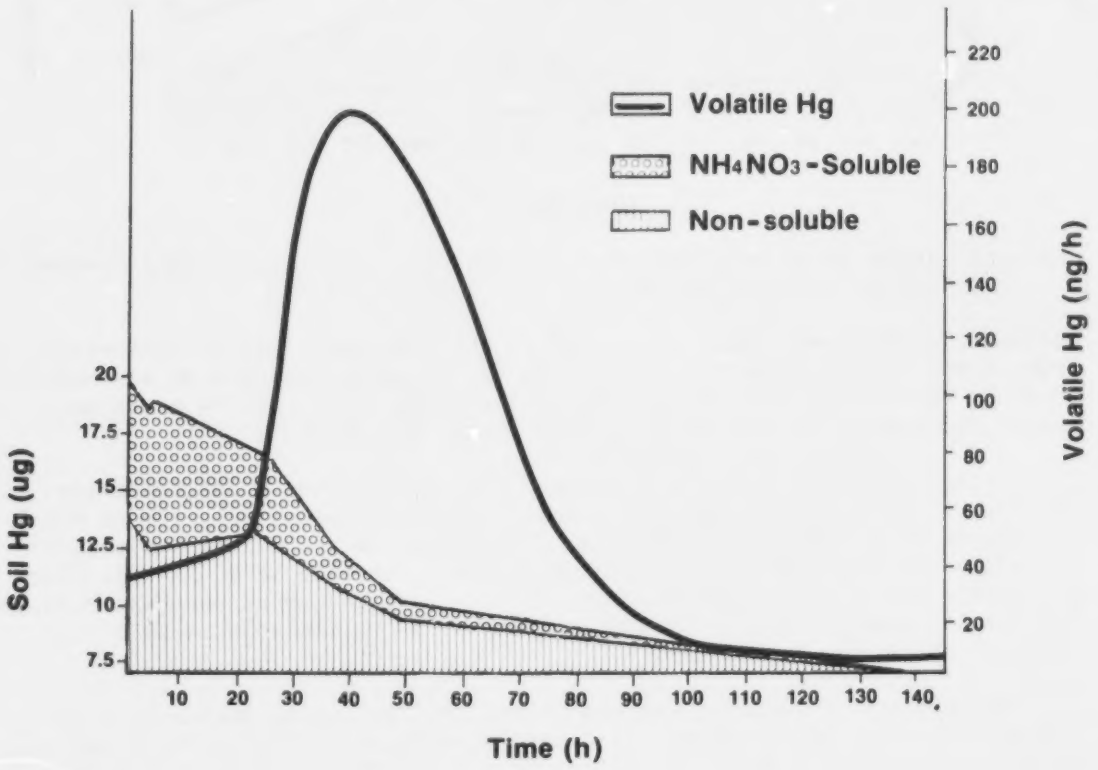

Figure 2. Rate of Hg volatilization and Hg remaining in sandy soil amended to 1 ppm Hg. The soil Hg (µg) axis is nonzero.

Sandy soil was also amended with $Hg(NO_3)_2$ at a concentration of 10 µg Hg/g soil (10 ppm Hg). During the 6-day incubation period, approximately 50% of the applied Hg was lost in the vapor form (Figure 3). There was a lag time of some 20 hours before the onset of rapid volatilization, but in this case no distinct volatilization peak was noted, rather the maximum rate of between 700 and 900 ng Hg/h persisted for some 50 hours before

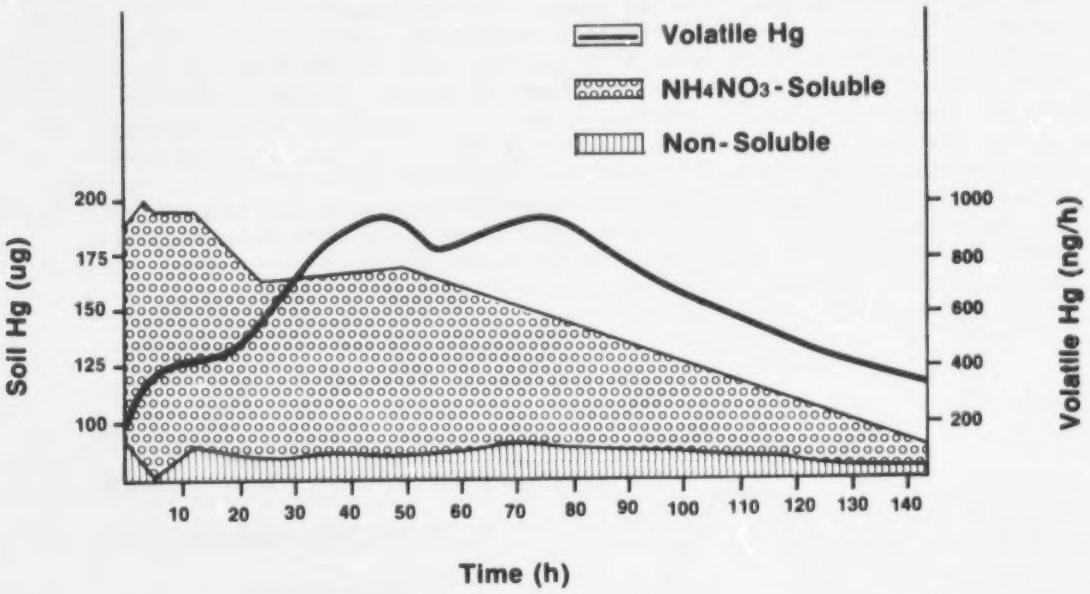

Figure 3. Rate of Hg volatilization and Hg remaining in sandy soil amended to 10 ppm Hg. The soil Hg (µg) axis is nonzero.

gradually decreasing. Even at the end of the test there was a substantial rate of volatile Hg loss. A decrease in the 1N NH_4NO_3-soluble Hg was noted after 12 hours and continued throughout the study (e.g., at 144 hours only 16% of the applied Hg was still soluble versus 50% at the start).

Steam-autoclaved sandy soil amended with $Hg(NO_3)_2$ at a concentration of 20 µg Hg/20 g soil (1 ppm) had a total volatile loss of only 10% of the applied Hg after 144 hours (Figure 4). There was no initial lag in volatilization as with the non-sterile sand trials. The rate of Hg loss decreased rapidly during the first 16 hours and then remained about constant throughout the duration of the test. The decrease in 1N NH_4NO_3-soluble Hg during the test period was negligible.

The pattern of Hg loss from the clayey soil was quite different from that of the sandy soil. Clayey soil amended to a concentration of 1 ppm Hg as $Hg(NO_3)_2$ lost a total of 20% of the applied Hg during the 144-hour examination period (Figure 5). Of this amount, 80% of the volatile loss occurred within the first 36 hours. Since there was no 1N NH_4NO_3-extractable Hg from this soil, the loss of Hg was from the non-extractable fraction. In addition there was no lag period between Hg applications and peak Hg volatilization rate with any of the treatments of this soil.

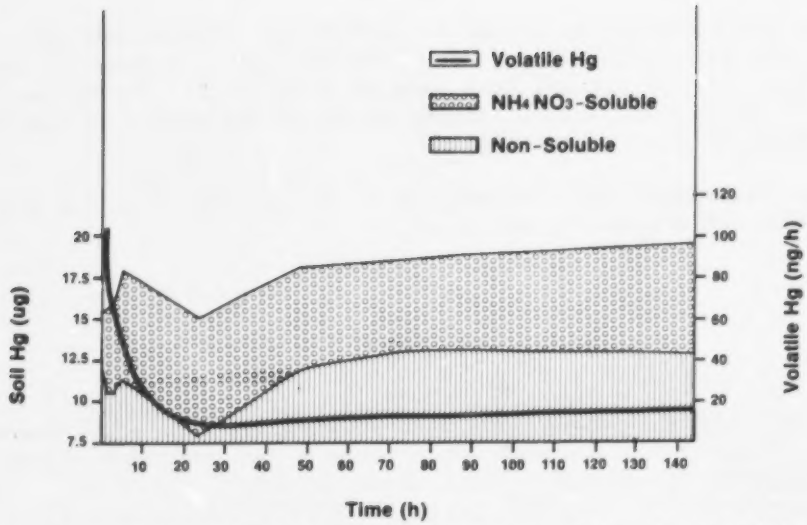

Figure 4. Rate of Hg volatilization and Hg remaining in sterilized sandy soil amended to 1 ppm Hg. The soil hg (µg) axis is nonzero.

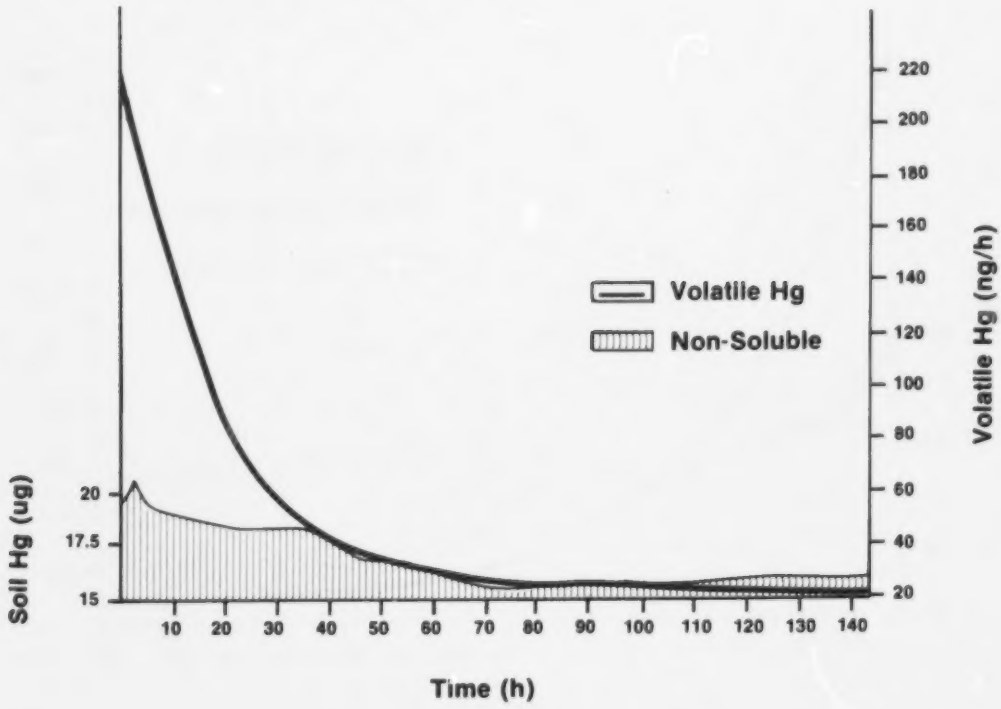

Figure 5. Rate of Hg volatilization and Hg remaining in clayey soil amended to 1 ppm Hg. The soil Hg (µg) axis and the volatile Hg (ng/h) axis are nonzero.

Clayey soil amended to 10 ppm Hg as $Hg(NO_3)_2$ volatilized 31% of this Hg during the test period (Figure 6). The bulk of this loss was during the first 3 days with an initial loss rate of 1,500 ng/h. During the first 24 hours of incubation, a small amount (4.5% of the total) of the Hg was found to be 1N NH_4NO_3-extractable.

Steam-sterilized clay lost only 2% of the applied 20 µg Hg (Figure 7). No 1N NH_4NO_3- extractable Hg was found.

DISCUSSION

The data from this study show that Hg was lost from soils amended with $Hg(NO_3)_2$. In every case, except one (Figure 3), almost 100% of the volatilization of applied Hg occurred within the first week. There was a considerable difference between the sand and clay. In general, the volatilization from clay was faster to start but less of the Hg was lost overall. Increasing the Hg concentration from 1 ppm to 10 ppm increased the loss of Hg from the sand by a factor of 11 and the clay by 15 times (Figures 3 and 6).

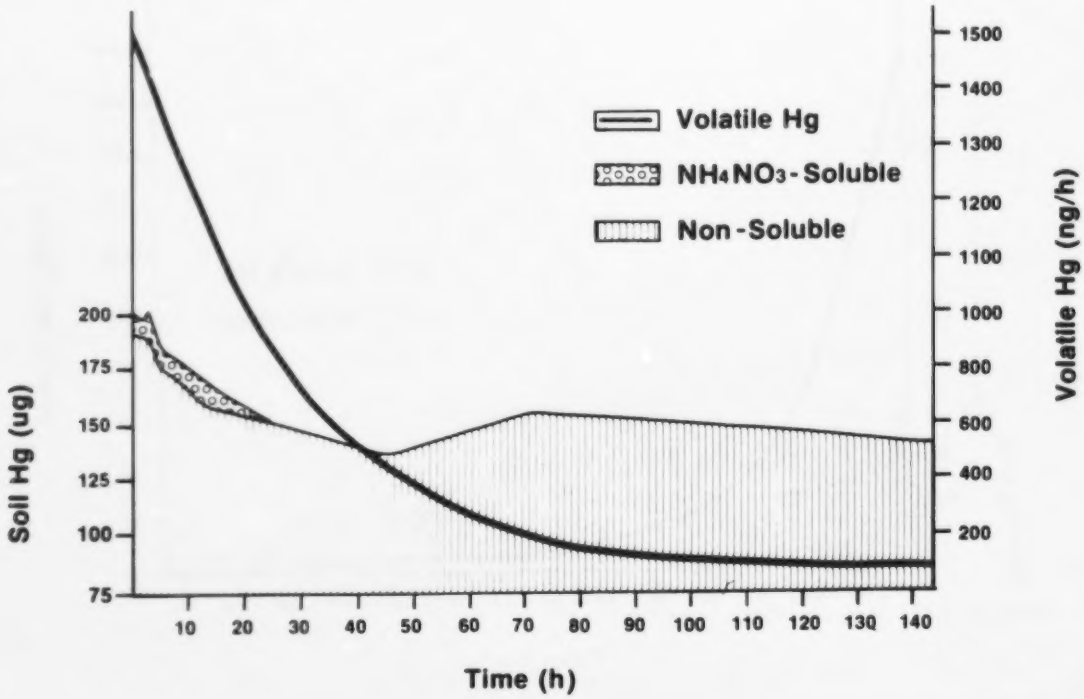

Figure 6. Rate of Hg volatilization and Hg remaining in clayey soil amended to 10 ppm Hg. The soil Hg (µg) axis is nonzero.

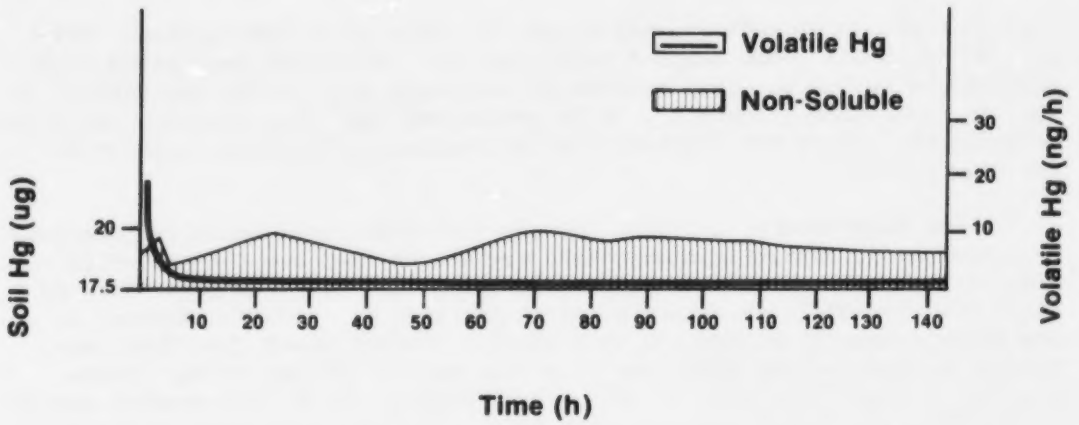

Figure 7. Rate of Hg volatilization and Hg remaining in sterile clayey soil. The soil Hg (µg) axis is nonzero.

Autoclaving the soils had a pronounced effect on the amount of Hg lost over the duration of these tests (144 hours, Figures 4 and 7). In the case of the sandy soil, this reduction was from 8.6 µg Hg to 2 µg Hg and a change of 4 µg Hg to 0.4 µg Hg for the clay. To insure that the Hg loss was initiated biologically, both soils were inoculated with 1 g of each of their respective nonsterile soils after 160 hours of incubation. No effect was noted with the clayey soil but within 8 hours the inoculated sandy soil started volatilizing Hg at a high rate (Figure 8). The volatiliza-

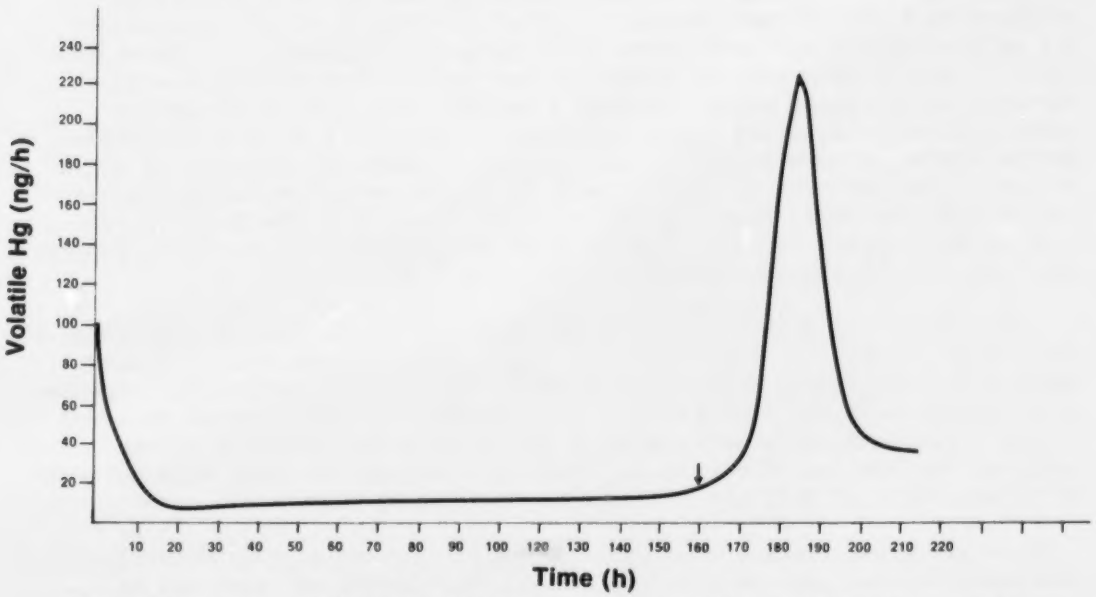

Figure 8. Rate of Hg volatilization from sterile sandy soil. Arrow indicates when soil was inoculated with 1 g of nonsterile soil.

tion from the sandy soil reached a peak 22 hours after inoculation. Total Hg loss after 214 total elapsed hours was 31%. Since the pool of 1N NH_4NO_3-extractable Hg had not been reduced in the sandy soil during the period of sterile incubation (Figure 4), it is speculated that this fraction was being volatilized. There was apparently no Hg available for volatilization in the clayey soil.

These data clearly indicate that Hg loss from these soils was mediated by a biological system, although this may not be the case for all soils since it has been shown that humic acid solutions can mediate the loss of ionic mercury (Hg^{++}) as elemental Hg (Alberts et al., 1974). Workers in the area of aquatic Hg cycling have shown in recent years that there are several microorganisms which can cause the volatilization of Hg. These microbes include *Escherichia coli, staphyloccus aureus,* and several species of *Pseudomonas* (Summers and Sugarman, 1974). Mercury loss from cultures of *Pseudomonas* amended with Hg^{++} was found to be elemental Hg (Tonomura, Furukawa, and Yamada, 1972). Some algae are also capable of causing the volatilization of Hg from solutions amended with $HgCl_2$ (Ben-Bassat and Mayer, 1975). The consensus is that these organisms cause Hg loss by the reduction of Hg^{++} to elemental Hg which is then lost because of its elevated vapor pressure (Tonomura, Maeda, and Futai, 1968; Izaki, 1977). It should be noted that while the pathways for volatile Hg in these soils are biological, the transformation of Hg^{++} into methyl Hg is abiological (Rogers, 1977, 1976). Methylmercury also has an elevated vapor pressure and, when formed one molecule at a time, it too is suspected of being volatile.

In the clayey soil the volatility rate continuously decreased while in the sandy soil the rate remained relatively stable for a few hours, followed by a significant increase. Since the enzyme system responsible for volatilization has been shown to be inducible (Furukawa and Tonomura, 1971) it may be that such an induction was responsible for the increases observed in the sandy soils (Figures 2 and 3). The lack of a similar pattern in the clayey soils may indicate the lack of a similar inducible enzyme system or more probably, the removal of mercury from microbe availability. The presence of only a small 1N NH_4NO_3-extractable fraction in the 10-ppm treatment (Figure 5) and no detectable 1N NH_4NO_3-soluble fraction in the 1-ppm treatment (Figure 4) of the clayey soils may also represent the lack of availability.

The number of soil microbes at the time of dosing were 30×10^6/g soil and 2×10^6/g soil for the clayey and sandy soils, respectively. These numbers did not change significantly after 30 hours of incubation. Species of organisms were not ascertained. The initial volatility rates observed in the clayey and sandy soils were in approximate relationship to the ratio of the numbers of microbes. However, insufficient data were collected to ascertain if this was a cause-effect relationship.

In all cases (except sand with 10 ppm Hg) the rate of Hg volatilization decreased to near zero long before all the applied Hg was used (80 to 71% remaining in clay and 50 to 57% remaining in sand). In the sandy soil the decrease in volatility correlated with a decrease in the amount 1N NH_4NO_3-extractable Hg and to a lesser extent with a decrease in the nonsoluble

fraction (Figures 2 to 4). However, since the volatilization of Hg decreased to near zero when the extractable Hg was exhausted, it is speculated that, in the case of the sand, Hg was lost only from the soluble fraction and the easily exchangeable fraction of the nonsoluble Hg. Data from studies on the volatile loss of Hg from water also link volatility with Hg availability. It has been shown that, as the concentration of complexing agents such as chlorine and bromine increase, the amount of Hg volatilized decreases because of a decrease in microbially available Hg (Jenne and Avotins, 1975). In other work with nonsterile solutions, it was shown that the amount of Hg volatilized from the solution depends on the amount of Hg which has become unavailable for microbial action due to combination with organic matter (Avotins and Jenne, 1975). The same authors reported that volatilization had almost ceased after 96 hours. These data are comparable with the findings from the sandy soil as far as the effect of Hg-complexing material, biological interactions on Hg volatilization, and the time required for the volatilization of the biologically available Hg. Note that during the time course of the sterile sand study, the extractable Hg content remained unchanged (Figure 4). Results from this study also support the hypothesis that the decrease in Hg volatility with time is caused by a loss of available Hg and not because the Hg is becoming more tightly bound with time.

While the volatilization of Hg from the clayey soil is also obviously a biological phenomenon (Figure 5 and 7), the Hg being volatilized was not correlated with a 1N NH_4NO_3-extractable source. Even though the Hg was not 1N NH_4NO_3-extractable, some 20 to 31% of it was biologically available (Figures 5 and 6). This would indicate that the Hg was initially in a form available to microbes but not in solution or easily removed from the soil exchange sites. How the Hg is being bound is not known but it has been shown that Hg is tightly complexed on organic matter and cannot be replaced with another ion except Hg (Strohal and Huljev, 1971). Since the clayey soil was higher in organic matter than the sandy soil, the soil organic matter could also be a viable factor in Hg retention by the clayey soil. The same could also be true for the inorganic exchange sites. Because the experiments were not designed to isolate clay or organic matter as factors affecting volatilizations, it is not possible from these data to emphasize which has the greater effect. It is possible that the amount of observed Hg volatility is not a true indication of the total Hg vapor occurring in the soil. This is because both clay and organic matter can sorb vapor-phase Hg (Krenkel, 1974; Trost and Bisque, 1971).

The data from both the sandy and clayey soils show that the amount of Hg volatilized from a soil is not an indication of the total amount of Hg contained by the soil. Conversely, knowing the total amount of Hg contained by a soil will not give an indication of potential losses due to volatilization. But, with more information from different soils some idea on how Hg is being bound by soils could be obtained from determining what portion of the total soil Hg is being volatilized.

The increase in volatility in response to the increase in Hg concentration (Figures 3 and 6) indicates that the organisms responsible for volatilization are capable of processing large quantities of Hg. It was apparent

that the Hg-binding sites in the clayey soil had not been saturated since over 150 µg of the initial 200 µg of Hg remained after volatilization had almost ceased. With the sandy soil, even after 170 hours (not shown in Figure 3), Hg was being evolved at a rate of 260 ng Hg/h. If evolution were to continue at this rate for 2 more weeks, 95% of the applied Hg would be volatilized. This would bring the remaining Hg to about the same concentration as that found in the sand amended with 10 µg of Hg after it ceased volatilization (11 µg).

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